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PROPERTIES OF CARBONATE ROCKS AFFECTING SOUNDNESS OF AGGREGATE

—A Progress Report

R. D. Harvey, G. S. Fraser, and J. W. Baxter

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ABSTRACT

The sodium sulfate soundness test (ASTM C88) of crushed stone is one of the principal tests for acceptance of aggregates for use in concrete highway construction in Illinois. A general study was made of 122 coarse aggregate samples, and 20 of these were chosen for a more detailed study. Correlations of the physical, chemical, and petrographic properties of carbonate rocks with their soundness loss were derived by statistical and graphical methods.

Indentation hardness decreases significantly with increases in soundness loss. Increasing water absorption correlates with increasing soundness loss for limestones and dolomites when the alumina content of the samples is low (< 0.90%). Alumina and absorption appear to be additive in their effects on soundness. Dolomite content correlates with soundness in a less significant manner. Graphical trends indicate increasing soundness loss correlates with increasing dolomite for limestones and with decreasing dolomite for dolomite rocks. Mean pore size showed little or no correlation with soundness loss.

Step-wise multiple regression analyses of the data give a correlation coefficient of 0.85 for the dolomites and one of 0.83 for the limestones. The standard error of the regression estimate of soundness losses is 5.2 percentage points for dolomite and 8.9 percentage points for limestone.

Microscope studies reveal a laminated or partly laminated structure of aggregate particles contributes significantly to high loss of soundness.

INTRODUCTION

The sodium sulfate soundness test (ASTM C88) is the test used in Illinois to judge the resistance of a sample of aggregate (mainly crushed limestone or dolomite) to deterioration under freeze-thaw conditions. The test involves alternately soaking a weighed sample of graded aggregate in an aqueous solution saturated with sodium sulfate (Na_2SO_4) and drying it in an oven. After a selected number of cycles, the sample is thoroughly washed, sieved to remove small broken pieces, dried, and weighed. The soundness loss is the weight loss expressed as a percentage.

The test is designed to accelerate the splitting, flaking, and crumbling that occurs when unsound aggregates are periodically exposed to freezing weather. The growth of sulfate crystals in the pores of the rock during drying exerts a force that is thought to be similar to that exerted by the growth of ice crystals. It is, however, more severe and tends to accelerate the breakup of aggregate particles. In similar tests, aqueous solutions of magnesium sulfate, sodium chloride, or alcohol are used. The tests are thought to be indicative of freeze-thaw durability.

Experience with use of the sodium sulfate soundness test in Illinois has been generally good. The maximum acceptable soundness loss for aggregates that are to be used in concrete pavements and bridges in Illinois is 15 percent during five cycles of the test. The test results are expressed as a percentage loss; therefore, a high numerical test result indicates poor durability.

The soundness test requires a large sample. For an aggregate that contains particles larger than 1½ inches, a sample of 7800 grams (17.2 lb) of a specific gradation is needed for the C88 test. To obtain the required weight of each of four sizes, nearly 110 kilograms (50 lb) of rock must be collected from the field or from cores for each laboratory test performed. In testing aggregates produced from operating quarries, the size of the required sample presents no particular problem, but in testing samples from selected rock strata, from outcrops, prospect pits, and drill cores, the amount of stone required is excessive. Further, tests on cores of carbonate rock strata crushed in the laboratory frequently give lower soundness losses than commercially produced aggregates from the same strata after a quarry has been opened up on the site (Richard Kiel, personal communication, 1969).

For these and other reasons, acceptance of quality aggregates for Illinois highway construction is not based on tests of ledge rock or drill cores. Only after the quarry or mine has been developed and the stone commercially crushed and screened will contracts be let for highest quality aggregates to be used in portland cement concrete construction. Because much crushed stone in Illinois is used for road and bridge construction, the fundamental properties of the stone that cause failure in the test should be determined, and these properties should be quantitatively related to test results so that accurate predictions of soundness can be made from measurements on relatively small samples.

The problem of evaluating the soundness of rock by testing relatively small samples from prospective quarry or mine sites will become increasingly acute as greater quantities of aggregates for construction are demanded and areas of supply decrease because of urban expansion and restrictive zoning. Our studies

seek to determine properties or indices of limestones and dolomites that can be used as reliable guides in prospecting for durable aggregates in the state.

Previous experience and test results have produced some helpful ideas. Woolf (1928) observed a correlation between the absorption and soundness loss (sodium sulfate) of sedimentary rocks. Studies of natural sands by Adams and Pratt (1945) and Mather (1947) showed that an increase in the sands' water absorption was very significantly correlated with an increase in their loss of soundness (magnesium sulfate solution). Verbeck and Landgren (1960) and Powers (1955), using direct freezing and thawing tests, showed that pore-size distribution, permeability, and degree of saturation had much influence on the freeze-thaw durability of aggregates. Dunn and Hudec (1966) concluded from a study of the disintegration of stone particles during water-soaking and oven-drying that subfreezing temperatures are not necessary to disrupt carbonate rocks. Their latest work (Dunn and Hudec, 1972) supports this conclusion and goes on to state that clay (largely illite) is the apparent common denominator in all the water-sorption sensitive rocks they tested, particularly the dolomitic rocks.

Experience in Illinois suggests that clayey, or argillaceous, material in a limestone contributes greatly to high loss in the soundness test. An earlier study on this project (Baxter and Harvey, 1969) showed a good correlation (correlation coefficient, 0.79) between alumina content and sulfate soundness for a limited number of drill core samples of fine-grained limestones and a few dolomites from the upper part of the St. Louis Limestone, Madison County, Illinois. The present study is an expansion of the previous one and includes results from 122 samples of carbonate rock taken from commercially operating quarries in Illinois by Illinois Division of Highway personnel.

Acknowledgment

The samples investigated for this study and the soundness and water absorption test results given in this paper were provided by personnel in the Bureau of Materials and Physical Research, Illinois Department of Transportation, Springfield, Illinois. We appreciate the cooperation of the Bureau in this study, especially that of Richard Kiel.

SAMPLES AND TEST RESULTS

The samples studied for this report are representative splits of coarse aggregates from the Bureau of Materials and Physical Research: gradation CA 5 (95 percent plus one-half inch), CA 7 (55 percent plus one-half inch, 95 percent plus 4 mesh), and CA 6 (55 percent plus 4 mesh, 75 percent plus 16 mesh). The soundness and water absorption test results on the samples are given in table 1, along with results of mineralogical and chemical tests. The samples are listed by number in the table in order of increasing geologic age. Classification by geologic unit is helpful because in many cases the test results of more than one sample from the same unit in an area are nearly the same, making the unit name a useful guide to the physical properties of untested samples.

TABLE 1—SAMPLES AND TEST RESULTS

Sample number	Geologic unit*	Illinois county	Soundness loss† (%) (Na_2SO_4)	Absorption† (%) (water)	Mineralogy** (%) > 20	2 - 20	< 2	Dolomite	Chemical results (%) Al_2O_3	SiO_2
70-871	Omega (P)	Clay	9.7	2.5	CC,Q	C,DO	--	12.2	3.44	39.1
70-1698	Millersville (P)	Christian	9.4	1.5	CC	C	Q	0.0	1.85	2.17
70-879‡	Millersville (P)	Clark	11.2	5.0	CC	C,DO	Q	2.8	1.54	2.19
70-933	Millersville (P)	Fayette	13.2	1.6	CC	DO	C,Q	10.5	1.34	3.46
69-12265	Millersville (P)	Vermilion	4.4	0.9	CC	--	C,DO	1.0	0.17	0.35
69-13813‡	Shoal Creek (P)	Clinton	16.5	2.0	CC	DO,C,Q	--	16.0	2.06	7.44
69-11356	Shoal Creek (P)	Livingston	12.9	1.8	CC	--	C,DO	0.5	0.46	1.64
69-14337	Shoal Creek (P)	Livingston	16.0	2.2	CC	DO	Q,C	7.8	0.56	2.94
69-14338	Shoal Creek (P)	Livingston	10.9	1.5	CC	DO	Q,C	7.8	0.05	0.44
70-2312	Shoal Creek (P)	Livingston	20.9	1.4	CC	C,DO,Q	--	3.8	1.72	7.64
70-2315	Shoal Creek (P)	Livingston	29.2	1.7	CC	C,Q	--	0.0	2.29	8.57
70-2316	Shoal Creek (P)	Livingston	13.1	1.5	CC	C	Q,DO	0.9	0.76	3.19
70-2427	Shoal Creek (P)	Livingston	15.8	1.5	CC	C,Q,DO	--	3.3	2.55	11.1
70-710	Shoal Creek (P)	Macoupin	12.9	2.8	CC	DO	C	16.9	0.62	0.68
69-11499	Shoal Creek (P)	Washington	11.0	1.9	CC	Q,DO,C	--	7.4	1.54	7.13
69-11500	Shoal Creek (P)	Washington	12.8	2.2	CC	Q,DO,C	--	11.8	2.71	9.26
69-14191	Lonsdale (P)	Menard	22.0	2.9	CC	DO	C	18.6	0.50	1.11
70-1590	Lonsdale (P)	Peoria	18.0	3.8	CC,Q	DO,C	--	10.8	2.38	26.0
70-191	Kinkaid (M)	Johnson	6.1	0.8	CC	Q,DO	C	4.0	0.74	10.60
70-192‡	Kinkaid (M)	Johnson	4.8	0.9	CC	Q,DO	C	4.9	0.77	8.82
70-197	Glen Dean (M)	Randolph	12.6	2.3	CC	--	C	0.0	0.50	0.72
69-10678‡	Fraileys (M)	St. Clair	36.3	1.6	CC	DO	C,Q	8.9	1.19	4.87
70-553	Ste. Genevieve (M)	Hardin	7.5	0.9	CC	Q,DO	C	13.8	0.49	5.45
70-558	Ste. Genevieve (M)	Hardin	2.5	0.6	CC	DO	C	8.6	1.04	2.22
70-1523	Ste. Genevieve (M)	Hardin	9.8	2.3	CC	DO	Q,C	1.2	0.41	2.12
70-1526	Ste. Genevieve (M)	Hardin	5.4	2.8	CC	DO	C,Q	1.5	0.75	2.05
69-10190	Ste. Genevieve & St. Louis (M)	Massac	1.8	0.7	CC	DO	Q,C	7.5	0.31	1.57
70-878‡	Ste. Genevieve & St. Louis (M)	Union	8.3	0.7	CC	DO,Q	C	6.4	1.19	9.15
70-1353‡	St. Louis (M)	Madison	3.6	0.6	CC	--	Q,C	0.0	1.01	1.50
70-1354	St. Louis (M)	Madison	5.4	0.9	CC	C	Q,DO	1.1	1.10	2.50
69-13986	St. Louis (M)	St. Clair	11.9	2.1	CC	DO,Q	C	8.1	0.78	7.93

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TABLE 1—*Continued*

Sample number	Geologic unit*	Illinois county	Soundness loss† (%) (Na_2SO_4)	Absorption† (%) (water)	Mineralogy** (%) > 20	2 - 20	< 2	Dolomite	Chemical results (%) Al_2O_3	SiO_2
69-13987	St. Louis (M)	St. Clair	3.8	1.2	CC	--	Q	0.0	0.18	2.97
70-1803	St. Louis (M)	St. Clair	1.3	1.0	CC	--	Q,C	0.0	0.31	3.39
70-1528	St. Louis (M)	St. Clair	8.6	1.4	CC	DO	C,Q	1.3	0.62	2.45
70-2003	St. Louis (M)	St. Clair	11.8	2.2	CC	DO	Q,C	6.7	0.61	3.21
69-10392‡	St. Louis (M)	Schuyler	15.6	3.5	CC,DO	Q,C	--	35.4	1.29	17.8
70-202	Salem (M)	Randolph	2.3	0.6	CC	--	--	0.0	0.07	0.18
70-3170	Harrodsburg (M)	Pulaski	6.4	1.8	CC	--	C,Q	5.2	0.64	2.38
70-1591	Warsaw (M)	Hancock	18.9	4.8	CC,Q	DO	C	7.2	0.55	37.4
70-1702	Keokuk-Burlington (M)	Adams	16.4	4.2	CC,Q	--	C	0.0	0.21	29.6
70-1970	Keokuk-Burlington (M)	Adams	13.8	4.2	CC,Q	DO	C	3.5	0.28	26.1
70-3134	Keokuk-Burlington (M)	Calhoun	12.8	3.1	CC	DO,Q	C	15.6	0.35	5.87
69-11864	Keokuk-Burlington (M)	Greene	11.6	2.8	CC	Q	DO	0.3	0.28	18.5
69-10834	Keokuk-Burlington (M)	Greene	7.3	1.2	CC	DO	Q,C	5.6	0.66	1.44
70-1358	Keokuk-Burlington (M)	Greene	5.5	1.3	CC	--	Q,C	0	0.44	2.02
70-1359	Keokuk-Burlington (M)	Greene	6.1	2.2	CC	Q	DO,C	0.3	0.24	3.81
70-2985	Keokuk-Burlington (M)	Hancock	31.1	4.9	CC,Q	DO,C	--	10.1	1.20	35.2
70-667	Keokuk-Burlington (M)	Henderson	33.3	7.3	CC,Q	DO,C	--	18.9	1.41	44.1
70-2984‡	Keokuk-Burlington (M)	Henderson	18.1	5.1	CC,DO	Q,C	--	27.5	1.18	17.6
69-10586	Keokuk-Burlington (M)	Pike	18.1	3.5	CC,Q	--	C,DO	0.3	0.48	35.2
70-1196	Keokuk-Burlington (M)	Pike	8.0	1.5	CC	Q	C,DO	3.1	0.76	7.29
70-1971	Keokuk-Burlington (M)	Pike	11.8	1.6	CC	Q	DO	0.8	0.16	6.64
70-2986	Keokuk-Burlington (M)	Warren	21.3	6.5	CC,DO	Q	C	31.9	0.96	20.4
69-11447	Cedar Valley (D)	Rock Island	11.1	2.2	CC	--	C,Q	8.7	0.58	1.82
69-10920	Wapsipinicon (D)	Rock Island	11.3	2.2	CC	--	C,Q	0.0	0.18	0.22
69-13467	Wapsipinicon (D)	Rock Island	4.6	1.2	CC	--	C	0.0	0.20	0.53
69-13468	Wapsipinicon (D)	Rock Island	4.0	2.1	CC	--	C,Q	0.0	0.26	0.42
69-10091	Racine (S)	Cook	13.3	--	DO	Q,C	--	82.9	1.57	8.95
69-11469	Racine (S)	Cook	7.6	2.0	DO	Q,C	--	82.9	1.74	11.2

* Geologic names are abbreviated as P = Pennsylvanian, M = Mississippian, D = Devonian, S = Silurian, and O = Ordovician.

† Determined by Illinois Division of Highways, Springfield, IL.

** Code for minerals: CC is calcite, DO is dolomite, Q is quartz, C is clay. Dash indicates none detected. The indicated percentages are best approximations.

‡ Additional data on these samples are given in table 4.

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TABLE 1—*Continued*

Sample number	Geologic unit*	Illinois county	Soundness loss† (%) (Na_2SO_4)	Absorption† (%) (water)	Mineralogy** (%) > 20	2 - 20	< 2	Dolomite	Chemical results (%) Al_2O_3	SiO_2
70-2511	Racine (S)	Cook	11.5	2.7	DO	C,Q,CC	--	71.9	2.18	19.3
70-357	Racine (S)	Cook	3.2	1.5	DO	Q	C	87.6	0.57	7.58
70-760	Racine (S)	Cook	6.7	1.8	DO	Q,C	--	90.4	1.86	4.93
70-762	Racine (S)	Cook	7.1	1.4	DO	Q,C	--	91.7	1.62	5.09
70-1406‡	Racine (S)	Cook	1.6	1.4	DO	--	Q	87.1	0.04	1.87
69-11448	Racine (S)	Rock Island	2.4	5.6	DO	--	--	99.0	--	--
69-10089	Racine-Joliet (S)	Du Page	7.6	2.3	DO	Q,C	--	86.6	1.39	8.65
70-820‡	Racine-Joliet (S)	Du Page	11.0	3.0	DO	Q,C	--	85.2	1.23	8.17
70-822	Racine-Joliet (S)	Du Page	8.0	2.8	DO	Q,C	--	84.2	1.65	7.66
69-11863	Joliet (S)	Jersey	9.5	2.3	CC	Q,DO	C	5.1	0.88	17.24
70-1356‡	Joliet (S)	Jersey	4.1	5.2	DO	C,Q	CC	94.8	1.39	1.98
70-3136	Joliet (S)	Jersey	8.6	2.5	CC	Q,DO	C	3.0	0.85	21.1
69-13246	Joliet (S)	Kankakee	10.6	2.2	DO	Q,C	--	83.2	1.03	9.78
69-13247	Joliet (S)	Kankakee	9.0	2.5	DO	Q,C	--	81.6	1.66	11.0
69-13248	Joliet (S)	Kankakee	8.7	2.5	DO	Q,C	--	82.0	1.91	9.67
69-13249	Joliet (S)	Kankakee	9.4	2.1	DO	Q,C	--	60.5	1.70	9.41
69-13250	Joliet (S)	Kankakee	9.0	2.2	DO	Q	C	81.1	0.59	10.7
69-13251	Joliet (S)	Kankakee	6.8	2.5	DO	Q,C	--	84.7	1.51	8.87
69-13699	Joliet (S)	Kankakee	8.4	2.1	DO	Q,C	--	81.1	2.16	10.8
69-13700	Joliet (S)	Kankakee	8.9	2.0	DO	Q,C	--	77.0	2.52	12.3
69-13701	Joliet (S)	Kankakee	8.7	2.1	DO	Q,C	--	81.5	2.65	10.8
70-126	Joliet (S)	Kankakee	6.5	2.9	DO	Q,C	--	87.4	1.31	6.68
70-1410	Joliet (S)	Will	6.4	1.9	DO	Q,C	--	86.5	1.39	3.69
70-1960	Joliet (S)	Will	13.7	1.3	DO	Q,C	--	83.7	1.13	7.35
70-1961‡	Joliet-Kankakee (S)	Will	16.4	2.8	DO	Q,C	--	82.7	1.26	9.43
70-506‡	Kankakee (S)	Kane	8.9	1.9	DO	Q	C	89.8	0.50	6.98
70-507‡	Kankakee (S)	Kane	8.5	2.0	DO	Q,C	CC	81.7	0.88	5.47
70-128	Kankakee-Edgewood (S)	Whiteside	8.5	2.7	DO	Q	C	86.8	0.55	7.45
70-13‡	Edgewood (S)	De Kalb	11.3	2.6	DO	Q	C	86.2	0.57	9.98
70-15	Edgewood (S)	De Kalb	10.0	2.9	DO	Q	C	86.9	0.72	8.75
69-11048	Galena (O)	Boone	9.0	2.1	DO	--	C,Q	85.5	0.85	1.49
70-609	Galena (O)	Jo Daviess	9.4	3.0	DO	--	C	94.8	0.75	1.42
70-2043	Galena (O)	Kane	11.2	1.4	DO	C,CC,Q	--	85.2	1.16	5.67
70-2051‡	Galena (O)	Kane	4.8	1.2	DO	C,CC,Q	--	82.6	1.11	5.31
70-330	Galena (O)	Kendall	10.7	1.7	CC,DO	--	C,Q	24.1	0.34	0.68
70-331	Galena (O)	Kendall	1.8	1.4	DO	--	C	92.2	0.24	0.56
70-332	Galena (O)	Kendall	8.7	1.5	CC	DO	C	20.0	0.34	1.09

(Continued on next page)

TABLE 1—*Concluded*

Sample number	Geologic unit*	Illinois county	Soundness loss† (%) (Na_2SO_4)	Absorption† (%) (water)	Mineralogy** (%) > 20	2 - 20	< 2	Dolomite	Chemical results (%) Al_2O_3	SiO_2
70-2562	Galena (O)	Ogle	5.5	2.4	DO	--	C	91.5	0.58	1.82
69-11373‡	Galena (O)	Stephenson	23.2	6.0	DO	Q	C,CC	84.5	0.59	6.35
69-12131	Galena (O)	Winnebago	11.7	3.2	DO	--	C,CC,Q	92.4	0.21	0.98
69-12132	Galena (O)	Winnebago	9.9	3.3	DO	--	C,Q	90.7	0.19	0.46
69-11673	Galena (O)	Winnebago	26.8	5.8	DO	Q	C	90.5	0.21	3.62
70-1043	Galena (O)	Winnebago	14.9	3.1	DO	Q,C	CC	87.0	1.13	3.88
69-12966	Galena-Platteville (O)	Ogle	23.6	3.1	DO	Q,C	--	82.3	1.90	8.60
69-12967	Galena-Platteville (O)	Ogle	19.9	2.2	DO	Q,C	--	84.6	1.41	5.75
70-3132	Platteville (O)	Calhoun	12.4	1.6	CC	DO	C,Q	16.6	0.80	2.65
69-14295	Platteville (O)	La Salle	6.0	2.0	DO	--	C,Q	94.6	0.55	2.64
70-101	Platteville (O)	La Salle	15.5	2.2	DO	--	C,Q	91.7	0.41	2.95
70-102	Shakopee (O)	La Salle	15.6	4.2	DO	CC,Q,C	--	69.6	1.52	13.2
OUT OF STATE SAMPLES STUDIED										
69-13244	Indiana		8.3	1.2	DO	CC,C	--	60.5	1.82	2.03
69-13245	Indiana		3.6	1.6	DO	--	C,CC	81.6	0.86	1.35
70-123	Indiana		27.5	2.4	DO	C,CC,Q	--	76.8	1.22	6.05
70-3107‡	Indiana		17.5	2.3	DO	CC	C	87.9	0.64	1.36
70-600	Indiana		6.1	0.9	CC	--	Q,C	1.5	0.63	3.84
70-657‡	Indiana		9.2	0.9	CC	DO	Q	9.8	0.17	2.11
70-1198	Indiana		13.1	2.3	CC	Q,DO,C	--	14.0	1.01	9.54
70-1863	Indiana		6.4	0.9	CC	--	C	0.0	0.70	1.98
69-11539	Missouri		3.2	0.6	CC	--	--	0.0	--	--
70-2800	Missouri		0.8	0.5	CC	--	C	7.9	0.82	2.69
70-3167	Missouri		1.7	0.3	CC	DO	C	8.0	0.94	2.43
70-388	Iowa		11.0	4.2	DO	CC	C	80.7	0.37	0.63
70-1657‡	Iowa		2.2	2.0	DO	CC	C,Q	85.7	0.13	0.72
70-2352	Iowa		4.6	0.9	CC	DO,C	--	10.4	1.27	1.3

* Geologic names are abbreviated as P = Pennsylvanian, M = Mississippian, D = Devonian, S = Silurian, and O = Ordovician.

† Determined by Illinois Division of Highways, Springfield, Illinois.

** Code for minerals: CC is calcite, DO is dolomite, Q is quartz, C is clay. Dash indicates none detected. The indicated percentages are best approximations.

‡ Additional data on these samples are given in table 4.

The samples of aggregate received were split several times to reduce their bulk size. One of the final splits of each was pulverized for chemical and mineralogical analyses, and petrographic analyses were made on the other final split. All major chemical constituents were determined for each of the samples, although only the alumina (Al_2O_3) and silica (SiO_2) analytical results are reported in table 1. Results of all the analyses are on open file at the Illinois State Geological Survey, Urbana, Illinois. The major and minor mineral components in the sample were determined by X-ray diffraction analyses (table 1). All samples were classified as either limestones (mainly $CaCO_3$) or dolomites (mainly $CaCO_3 \cdot MgCO_3$), and they contain various amounts of quartz (SiO_2) and clay (complex aluminum silicates) as impurities. The dolomite composition of each sample (table 1) was calculated from the chemical analysis, and its presence was verified by X-ray diffraction when possible.

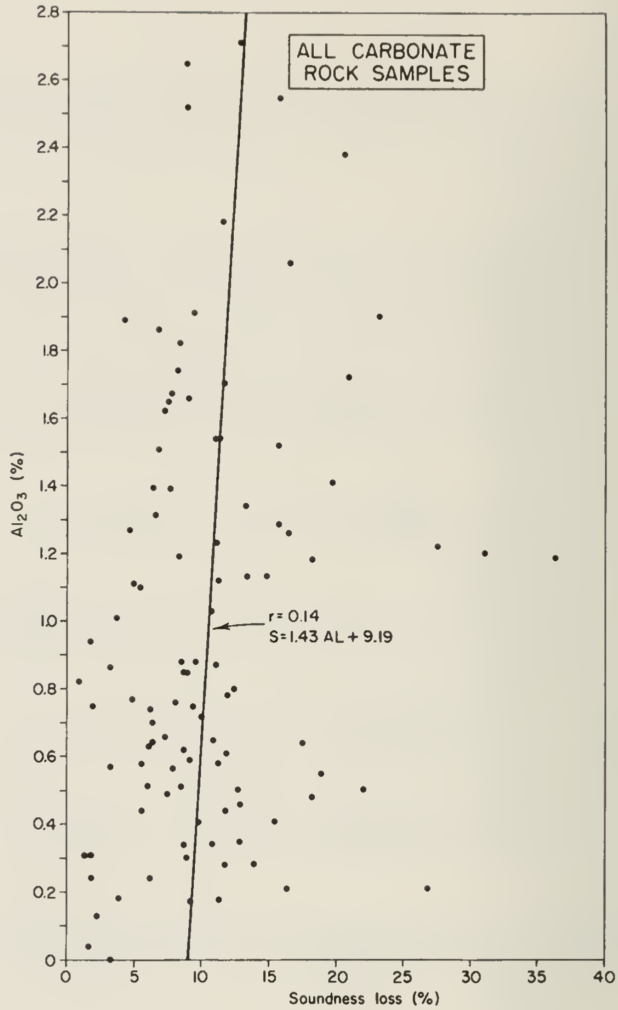

Fig. 1 - Alumina content (AL) versus soundness loss (S). Correlation coefficient, r, and linear regression equation for S, in terms of the independent variable, are given here and in similar later figures.

ANALYSIS OF RESULTS

Consideration of the previously cited literature suggested that certain properties of the aggregate, including dolomite content, water absorption, and alumina content, might possibly be used to explain the behavior of the samples in the soundness test. Graphs of these properties show a wide scatter of data points (figs. 1, 2, 3). The values of the correlation coefficients, r, are given on the graphs. The r value gives a measure of the scatter of the data points from the "best-fit line," the linear regression line. The mathematics of linear regression and correlation are discussed in most text books on statistics, for example Huntsberger's (1967, p. 255-274). We assume the properties selected for evaluation do indeed have some effect on the behavior of the rock; we therefore assume further that the correlation coefficient and its sign are indicative of the relative degree to which the variables affect soundness and of the direction of that effect.

A slight trend is evident in figure 1—as alumina content increases, soundness loss also rises. The correlation coefficient, 0.14, is very low, however, suggesting alumina (or clay content) has relatively little direct effect on soundness loss. This coefficient is much lower than the one (0.79) found between the same variables in the previous study by Baxter and Harvey

(1969). However, only a fairly restrictive type of limestone lithology—mostly dense and fine grained—was studied, and all of the samples were from the upper part of the St. Louis Limestone. The present study, on the other hand, includes a wide variety of carbonate rock types.

A plot of dolomite content versus soundness loss (fig. 2) shows the samples fall into two distinct groups, limestones and dolomites. Separate regression analyses of these groups show weak correlation trends. For the limestones, increasing dolomite content correlates weakly with increasing soundness loss. When limestone data points were plotted by geologic unit of the samples, that correlation trend was not evident. In the dolomite samples, increasing dolomite content correlates weakly with *decreasing* soundness loss. The dolomite samples of Silurian (table 1) geologic units generally showed a different trend than the Ordovician dolomites.

The regression line for water absorption plotted against sodium sulfate loss (fig. 3) has a positive slope, indicating that as water absorption increases soundness loss increases. The correlation coefficient is somewhat higher (r = 0.59) than those for alumina and dolomite contents, indicating that water absorption is possibly one of the main factors determining the performance of aggregate in the soundness test.

Classification by Petrographic Groups

Groupings of the data according to alumina and dolomite contents were made in an effort to reduce the scatter of points about the regression line that related percentage of water absorption to soundness loss. Histograms were made of the distribution of the percentage of alumina content and percentage of dolomite content to see whether the distribution of these two components was unimodal or whether it occurred in a

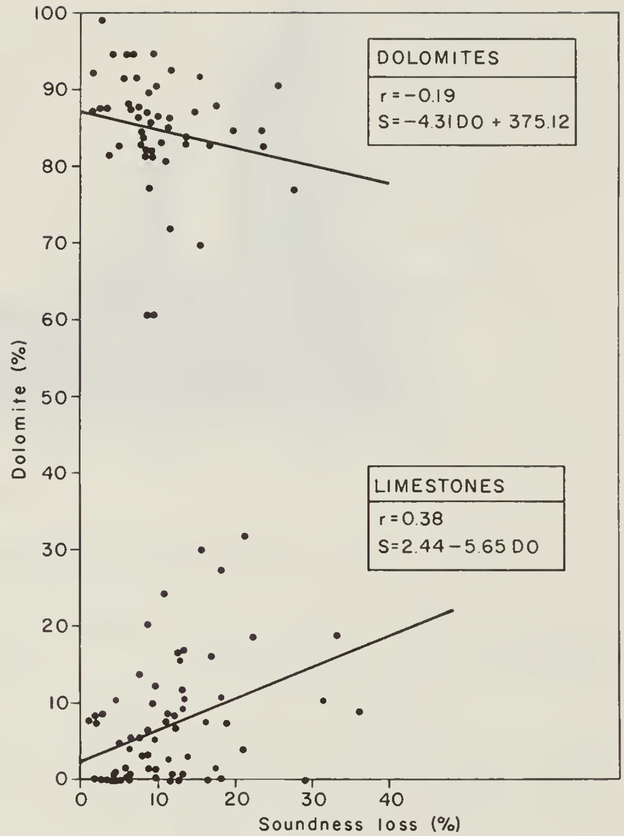

Fig. 2 - Dolomite content (DO) versus soundness loss (S).

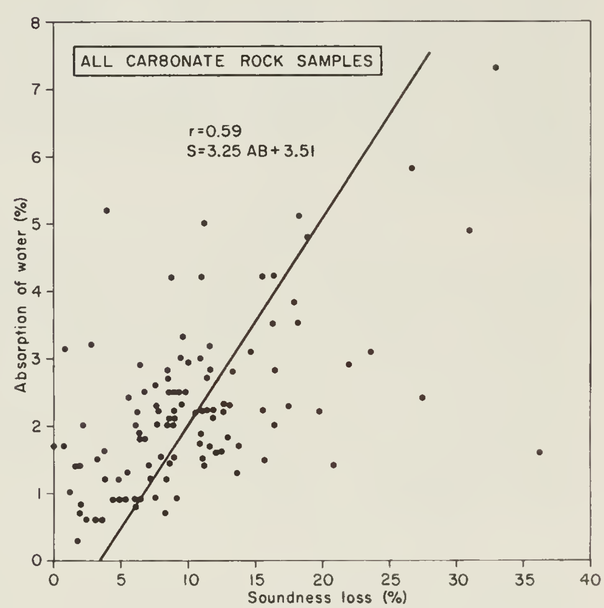

Fig. 3 - Absorption of water (AB) versus soundness loss (S).

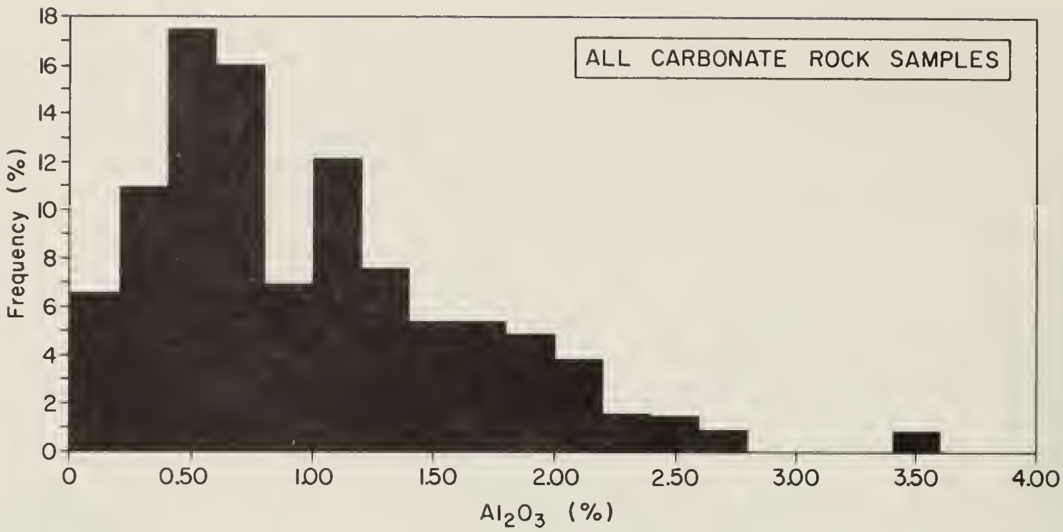

Fig. 4 - Frequency of the percentage of alumina in all carbonate rock samples.

polymodal distribution that might suggest natural groupings into which the data might be classified.

A break occurs in the alumina distribution near 0.9 percent (fig. 4). The samples were thus divided into two main groups: those with 0.9 percent or less alumina and those with more than 0.9 percent. We classified the former as low-alumina carbonate rocks and the latter as high-alumina carbonate rocks.

The histogram relating the percentage of dolomite mineral to its frequency of occurrence in the samples (fig. 5) shows all of the samples fell within two restricted ranges: 0 to 40 percent and 60 to 100 percent. The samples were thus further subdivided into two groups: those with 0 to 50 percent dolomite mineral, classified as limestones, and those with more than 50 to 100 percent dolomite mineral, classified as dolomites. Table 2 shows the soundness data grouped according to the two classes and their subdivisions and the mean and standard deviation of soundness loss of the classes.

To find out whether these groupings were legitimate, that is, whether the groups showed a significant difference in the results of their soundness loss, each group was tested against the others by means of a t-test. The t-test is a standard statistical procedure discussed in textbooks on statistics, such as Huntsberger's (1967).

A significance level of $\alpha = 0.10$ was chosen. Thus there would be a 0.90 probability that a significant difference existed between the two groups. At

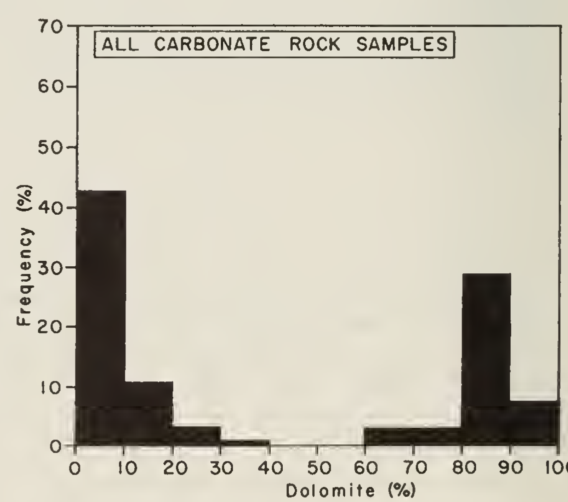

Fig. 5 - Frequency of the percentage of dolomite in all carbonate rock samples

this level, however, the test has a 0.10 probability of indicating a significant difference when none actually exists. The range of soundness, though, was small compared to the range in physical and chemical properties. This limited our ability to analyze the relations between soundness and physical and chemical properties because large changes in those properties have only a small numerical effect on the soundness results. It was decided, therefore, to risk the 0.10 probability of a false indication of significant difference in order to reduce the probability of failing to recognize a significant difference.

Table 3 shows the results of the t-test analyses of the soundness loss data for the low- and high-alumina carbonates, and for limestones versus dolomites in each. The calculated t-statistic (t_{cal}) between groups I and II is greater than the standard accepted value, (t_{val}), and, therefore, the differences observed between the two groups can be considered significant. This is suggested by comparison of the means of the two groups (table 2). The test failed to show any significant difference in the soundness loss between the

TABLE 2—GROUPINGS OF SOUNDNESS DATA

	I. Low-alumina carbonate rocks (0.00 - 0.90% Al_2O_3)					II. High-alumina carbonate rocks (0.90+% Al_2O_3)			
	Limestones			Dolomites		Limestones		Dolomites	
	7.3	1.8	11.3	3.6	9.9	8.3	31.1	8.3	6.5
	12.9	4.4	11.1	1.6	9.4	3.6	33.3	6.7	23.6
	8.0	1.3	9.5	8.5	8.9	5.4	16.5	7.1	15.6
	5.5	6.4	11.6	1.8	26.8	4.6	29.2	6.4	13.3
	11.8	2.3	6.1	6.0	3.2	36.3	15.8	13.7	11.5
	13.1	7.5	8.6	9.0	2.4	11.0	12.8	4.8	7.6
	0.8	4.8	9.8	9.0	10.0	13.2	18.0	7.6	4.1
	6.4	6.1	11.8	5.5	17.5	20.9	9.7	19.9	8.7
	8.6	6.1	12.6	15.5	11.0	9.4	2.5	10.6	8.4
	13.8	9.2	11.9	8.5	2.2	13.1	18.1	9.0	8.9
	3.2	18.1	16.0	11.3	23.2	15.6	21.3	9.4	8.7
	10.7	12.8	16.4	11.7		11.2	1.7	6.8	11.2
	8.7	18.9	12.9					11.0	14.9
	3.8	22.0	12.4					8.0	27.5
	10.9	4.0						16.4	
	5.4								
	4.6								
Mean	9.27					12.81			
Standard deviation	5.39					7.87			
No. samples	69					53			
Mean	9.20			9.41		15.11		10.90	
Standard deviation	4.82			6.50		9.68		5.46	
No. samples	46			23		24		29	

TABLE 3—RESULTS OF GROUPING ON BASIS OF ALUMINA CONTENT AND DOLOMITE CONTENT

Groups being compared	t_{cal}	t_{val}* (α = 0.10)†	Group differences
Low- and high-alumina carbonate rocks (I and II)	2.86	± 1.66	significant difference
Low-alumina limestones and dolomites (Ia and Ib)	0.15	± 1.67	no difference
High-alumina limestones and dolomites (IIa and IIb)	1.88	± 1.68	significant difference

* Standard accepted value, such as that from Huntsberger (1967, Appendix table III).

† Conclusions drawn with 10 percent chance of rejecting a true hypothesis.

low-alumina limestones and the low-alumina dolomites (note the similarity of the means of the soundness of these two subclasses in table 2). In this case, t_{cal} is numerically less than t_{val}. However, for the high-alumina carbonates, the limestones and dolomites show a significant difference. The high-alumina limestones have a mean soundness loss of 15.11 percent compared to 10.90 percent for the dolomites, indicating that relative abundance of alumina and dolomite are two of the properties of the samples that influence the soundness test results.

Graphical Analysis

Graphs were prepared for each of the three classes (low-alumina carbonates, high-alumina limestones, and high-alumina dolomites). The graphs relate soundness to apparent porosity, as measured by absorption of water (figs. 6, 7, and 8). The correlation coefficients and the equations of the least-squares regression lines for these sets of data are shown on the figures

Although the t-test showed no significant difference between low-alumina limestones and dolomites, the regression analyses between the absorption and soundness of the low-alumina carbonate samples (fig. 6) showed that the correlation of the two groups separately is slightly higher (r = 0.81 for limestones; r = 0.88 for dolomites) than when the groups are combined (r = 0.77). Apparently the t-test was not sufficiently sensitive to the slight difference that the two types of carbonates exhibit. The positive slopes of the regression lines indicate to us that, for low-alumina samples, water absorption has a direct effect on the performance of aggregate in the soundness test, causing increased loss as absorption increases. The correlation (r = 0.40) is not as good with high-alumina limestones (fig. 7), and a very poor correlation exists between absorption and soundness losses for high-alumina dolomites (fig. 8). This low correlation for the high-alumina carbonates is thought to be caused by the presence of clay laminations in some samples and their absence in others. Results pertaining to such laminations are discussed on page 17.

From the graphical analyses and t-tests it seems quite likely that clay content (measured as Al_2O_3) and porosity (measured as water absorption) are closely interrelated in their effect on soundness. The effects of increased water absorption may be enhanced by the presence of clay, or the effects of increased clay may be enhanced by the presence of water in pores of the rock. Soundness and alumina plus absorption for limestones and dolomites (fig. 9) have correlation coefficients of 0.70 and 0.49, respectively. The correlation coefficients of the soundness and alumina times absorption are smaller for both rock types. Thus, these data suggest the relation between the variables alumina and absorption is probably additive in terms of their effect on soundness, especially for limestones.

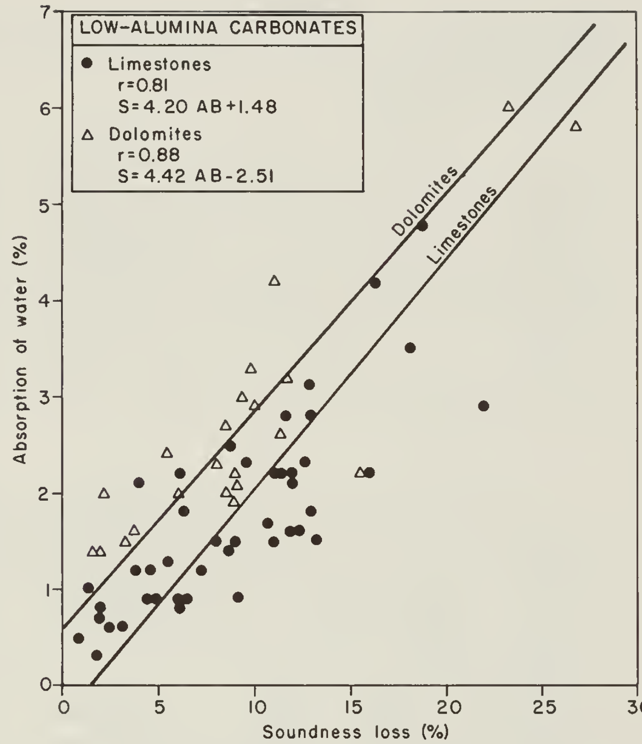

Fig. 6 - Absorption of water (AB) of low-alumina carbonate rocks versus soundness loss (S).

There is a considerable scatter of data points on the graphs illustrating these trends, and we suspect now that the three properties, alumina, dolomite content, and absorption, are not the only variables affecting the performance of the aggregates in the sulfate soundness tests. To test that belief, 20 representative samples were chosen from the three groups for further tests: five with data points above the trends shown in the graphs (figs. 6, 7, and 8) 10 with data points on the trends established, and five with data points below the trends. Table 4 lists the samples chosen.

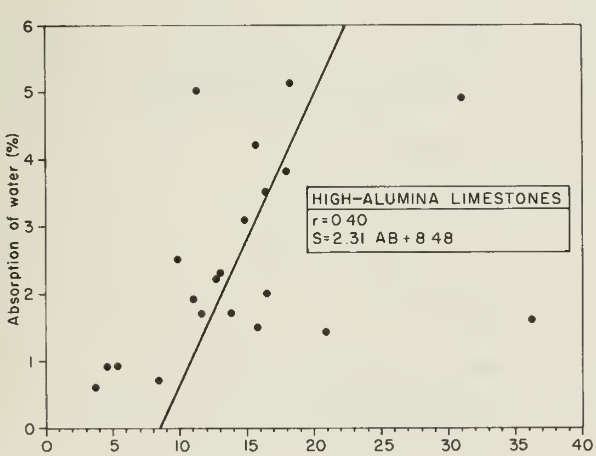

Fig. 7 - Absorption of water (AB) of high-alumina limestones versus soundness loss (S).

Hardness and Mean Pore Size

Two other variables were considered, hardness and pore-size distribution. The Rockwell hardness of the samples was measured by a Clark Hardness Tester with a ball indentor one-eighth of an inch in diameter and a load of 150 kilograms (Rockwell scale K). Twenty measurements were made on polished sections of representative pieces of aggregate, and the mean test result was taken as the hardness for the sample. The pore-size distribution was determined with a Quantimet Image Analyzing Microscope by techniques described by Harvey and Steinmetz (1971). This method detects and measures the length of chords across pores 0.9 or more microns (μm) in diameter on polished sections of the sample. The mean of the

chord lengths, measured from 30 or more microscopic fields of view, was taken to be characteristic of the size of pores in the sample.

The results of the analyses for hardness and mean pore size, along with other petrographic observations, are listed in table 4. The mean hardness for the limestones in relative and arbitrary units is 78.8, and for the dolomites it is 82.7. The mean of the mean pore size is 4.5 µm for the limestones and 6.2 µm for the dolomites.

Soundness loss was plotted against alumina, dolomite, and absorption of water for the 20 selected samples, and, as the same general trends and correlation coefficients were obtained for these samples as were obtained for the original samples (figs. 1, 2, 3), they are not shown here. Soundness loss plotted against hardness and mean pore size for the 20 samples is shown in figure 10.

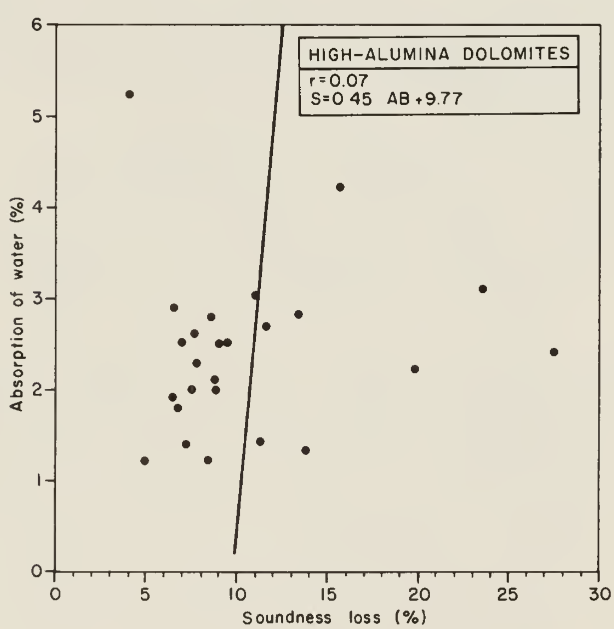

Fig. 8 - Absorption of water (AB) of high-alumina dolomites versus soundness loss (S).

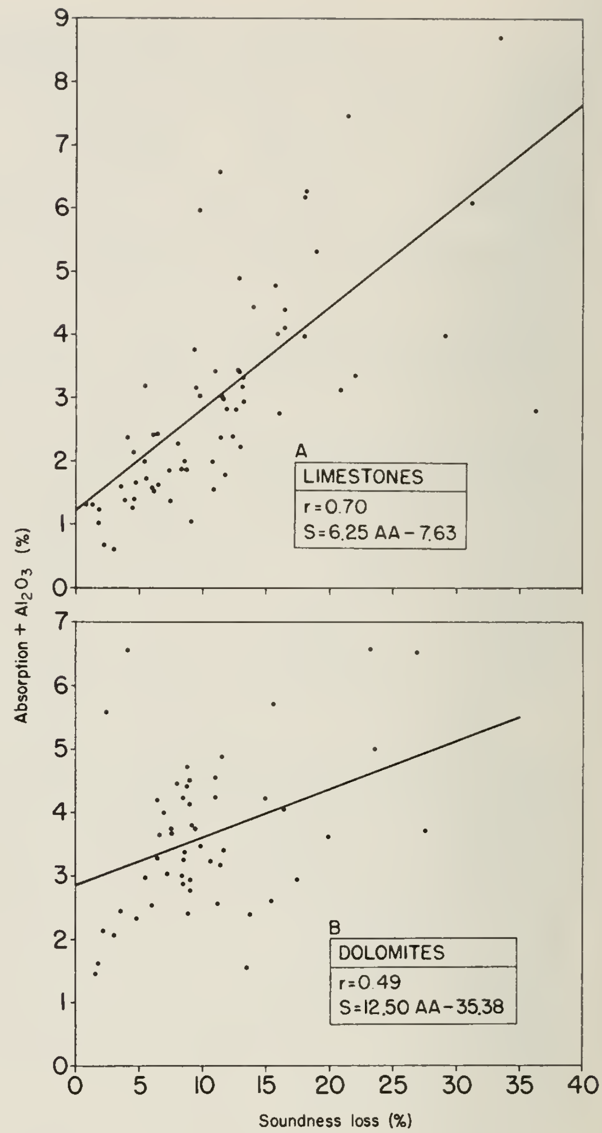

Fig. 9 - A. Absorption + alumina (AA) of limestones versus soundness loss (S).
B. AA of dolomites versus soundness loss (S).

A very significant correlation (r = -0.80) was observed between hardness of limestones and their soundness loss (fig. 10A) that indicates that hardness is a major property affecting the behavior of the aggregate in the soundness test. It is evident that the harder the stone the lower the soundness loss. Dolomites show (fig. 10A) the same significant correlation (r = -0.80). The rate of increase

TABLE 4—DETAILED ANALYSES ON SELECTED SAMPLES

Sample number	Soundness loss (%)	Classification of particles*	Color	Grain size	Structure	Hardness†	Mean pore size‡ (μm)	Absorption of water (%)
70-1406	1.6	Dol	gray	medium	isotropic	92.4	4.2	1.4
70-1657	2.2	Dol & Ls	gray & brown	variable	isotropic	80.4	3.0	2.0
70-1353	3.6	Ls	gray	fine	isotropic	85.6	5.5	0.6
70-1356	4.1	Dol	mixed gray	variable	isotropic	84.0	10.2	5.2
70-192	4.8	Ls	brownish gray	variable	isotropic	82.4	5.1	0.9
70-2051	4.8	Dol	light gray	variable	isotropic	90.3	4.8	1.2
70-878	8.3	Ls	mixed gray	fine	isotropic	78.5	3.7	0.7
70-507	8.5	Dol	light gray	medium	isotropic	81.7	7.3	2.0
70-506	8.9	Dol	light gray	variable	laminated	87.3	10.7	1.9
70-657	9.2	Ls	light gray	fine	isotropic	79.4	2.6	0.9
70-820	11.0	Dol	light gray	fine	isotropic	84.3	8.0	3.0
70-879	11.2	Ls	mixed gray	fine	partly laminated	76.6	3.4	5.0
70-13	11.3	Dol & Ls	gray	variable	laminated	79.9	6.4	2.6
69-10392	15.6	Ls	brownish gray	fine	partly laminated	76.6	7.7	3.5
70-1961	16.4	Dol	brownish gray	variable	partly laminated	81.8	2.6	2.8
69-13813	16.5	Ls	gray	fine	partly laminated	77.7	2.4	2.0
70-3107	17.5	Dol	brownish gray	variable	isotropic	76.7	5.5	2.3
70-2984	18.1	Ls & Dol	dark & light gray	variable	partly laminated	78.9	6.1	5.1
69-11373	23.2	Dol	brown	variable	isotropic	65.5	5.6	6.0
69-10678	36.3	Ls	mixed gray	fine	laminated	73.6	4.2	1.6

* Dol for dolomite, Ls for limestone.

† Rockwell scale K.

‡ The mean of the distribution of lengths of chords across pores, observed with a microscope, on carefully polished and random cross-sectional surfaces (Harvey and Steinmetz, 1971). The smallest pore resolved and measured was 0.9 μm in diameter.

of soundness loss with respect to decreasing hardness for limestones is more than twice that for dolomites. Mean pore size as we measured it, however, does not seem to have a significant effect on soundness (fig. 10B).

In an effort to characterize the combined effect of two or more of the variables on the soundness loss, several multiple regression correlation

experiments (tables 5 and 6) were made on the data. Each experiment involved different independent variables. The regression correlation analyses were performed in a step-wise procedure that allows the assessment of the relative rank that each independent variable has in maximizing the correlation or "goodness of fit." The intercept and coefficients given for each experiment define an equation of the estimate of soundness loss, S*. In experiment 1, for example (table 5):

$$S^* = 219.2 - 2.6\ H + 1.1\ P - 1.1\ AB - 0.04\ DO - 0.4\ AL,$$

where H is the hardness, P the mean pore size, AB the absorption of water, DO the dolomite content, and AL the alumina content of a sample of limestone for which a prediction of the soundness loss is desired. The subscripts to the coefficients given in table 5 indicate the rank of the variable as it is determined in the step-wise regression model. Hardness has the highest rank of 1 in each experiment, which indicates it is the most significant variable. Mean pore size ranks 2 for limestones but 5 for dolomites (table 6). Absorption ranks higher than alumina for limestones but lower than alumina for dolomites.

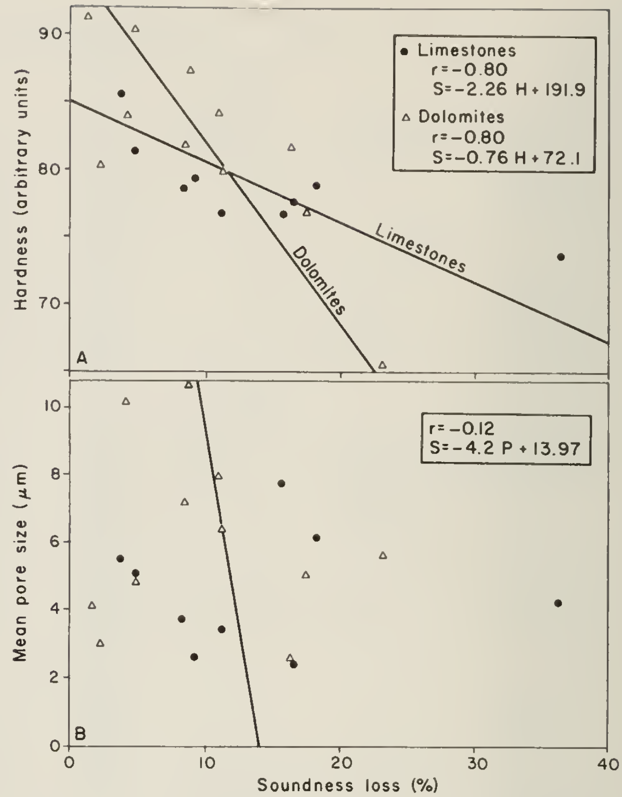

Fig. 10 - A. Hardness (H) of selected samples of limestones and dolomites versus soundness loss (S).
B. Mean pore size (P) of the selected samples versus soundness loss (S).

The best correlation coefficients are obtained when all five variables are included in the analysis—0.83 for limestones and 0.85 for dolomites. Some reduction in correlation occurs when variables are removed, except in experiment 4 for limestone (table 5) and experiment 2 for dolomites (table 6). It appears from these analyses, therefore, that one could consider only hardness and absorption in the evaluation of limestones and only hardness, alumina, and absorption in the evaluation of dolomites to obtain a prediction of soundness as good as one made when all the variables are considered. The regression equations predict the soundness loss (S) with a standard error of estimate of 4.3 to 8.9 percentage points. The standard error of estimate is obtained from $(S - S^*)^2/(n - 1 - k)$, where S is the observed (test result) and S* the predicted soundness loss, n the number of samples, and k the number of independent variables used in the regression equation.

For 17 out of the 20 samples, the regression equations predict which of the samples would be correctly judged as acceptable in Illinois (15 percent loss or less) and unacceptable (more than 15 percent). The regression analysis predicts the soundness loss of dolomites slightly more successfully than it does that of limestones.

TABLE 5—STATISTICAL ANALYSES OF DATA ON NINE LIMESTONE SAMPLES

MULTIPLE CORRELATION (Dependent variable is soundness loss, S)

Experiment	S intercept	Coefficient of independent variables and their rank (subscript)					Corr. coef., r	Standard error estimate of S
		Hardness	Mean pore size	Absorption	Dolomite	Al_2O_3		
1	219.2	-2.6_1	$+1.1_2$	-1.1_3	-0.04_4	-0.4_5	0.83	8.9
2	215.8	-2.5_1	X*	-0.5_2	-0.06_3	-1.7_4	0.82	8.1
3	200.7	-2.4_1	X	-0.6_2	X	-0.6_3	0.81	7.4
4	202.9	-2.4_1	X	-0.6_2	X	X	0.81	6.7
5	191.9	-2.3	X	X	X	X	0.80	6.3

* X indicates this variable is not considered in this experiment.

Rock Structure

Petrographic descriptions of the selected samples were made and organized on the basis of rock type (limestone or dolomite), color, relative grain size, and structure (table 4). The characterization of structure based on the presence or absence of parallel laminations or bands of component mineral grains was made by microscopic examination of polished sections. The samples that consisted entirely of nonlaminated stone particles were classified as isotropic; samples which had laminations within each particle were classified as laminated. Samples consisting of mixed nonlaminated and laminated particles were classified as partly laminated. For convenience, the soundness loss and absorption data from table 1 are repeated in table 4. The samples are listed in the table in order of increasing loss of soundness.

Seven out of nine samples with soundness losses greater than 11 percent have partly laminated or laminated structures (table 4); the two exceptions are characterized by having an extra-high absorption or extra-low hardness. One laminated sample (a dolomite with extra-large pores) had a soundness loss of less than 11.0 percent, but laminated structure in aggregate pieces is definitely a significant factor contributing to increases in soundness loss.

CONCLUDING REMARKS

The complexity and interrelation of properties that are thought to influence the soundness behavior of carbonate rocks are evident in this progress report. Hardness, absorption of water, alumina content, dolomite content, and laminated microstructure, which are the principal properties found to correlate with soundness, may be useful in predicting the sodium sulfate soundness of carbonate rocks that are to be used for aggregate. The following correlations

TABLE 6—STATISTICAL ANALYSES OF DATA ON ELEVEN DOLOMITE SAMPLES

MULTIPLE CORRELATION (Dependent variable is soundness loss, S)

Experiment	S intercept	Coefficient of independent variables and their rank (subscript)					Corr. coef., r	Standard error estimate of S
		Hardness	Mean pore size	Absorption	Dolomite	Al_2O_3		
1	98.9	-0.9_1	0.2_5	-1.0_3	-0.2_4	4.1_2	0.85	5.2
2	93.2	-0.9_1	X*	-1.0_3	-0.1_4	4.4_2	0.85	4.7
3	88.6	-1.0_1	X	-1.3_3	X	4.7_2	0.84	4.4
4	70.3	-0.8_1	X	X	X	3.2_2	0.82	4.3
5	72.1	-0.8	X	X	X	X	0.80	4.4

* X indicates this variable is not considered in this experiment.

of these properties with soundness loss became evident:

1. When hardness (measured by the Rockwell scale K on polished specimens) decreases, soundness loss increases.

2. When water absorption increases, soundness loss tends to increase. This is especially true for rocks that are relatively free of clay (alumina < 0.9 percent).

3. When dolomite content in limestones increases, the soundness loss tends to increase slightly. Conversely, when dolomite content in dolomite rocks increases, the soundness loss tends to decrease slightly. The dolomite content is most useful for classifying the sample as either limestone or dolomite rock, which must be evaluated separately.

4. When laminations, which may be observed in microscope studies of polished surfaces, occur within the aggregate particles, soundness loss is nearly always higher than 11 percent.

5. Clayey limestones (those with 0.9 percent or more alumina) have an average soundness loss twice that of limestones that are relatively free of clay. Clayey dolomites have soundness losses that average about the same as those for purer dolomites.

Although the information revealed in this report is useful, the properties studied do not predict soundness loss with sufficient accuracy, and further studies are being made. The highest correlation coefficient we obtained from our data by multiple correlation regression analysis was 0.85. In this study it would have been desirable to have a coefficient of at least 0.90. Insufficient precision in determinations of soundness loss and other properties and the possibility that our samples were not truly representative probably account for some of the residual error in the soundness predictions. Hardness tests and petrographic studies of the remaining samples to be studied are now in progress.

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